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IS 1800 (1989): Perfumery Material - Geraniol [PCD 18: Natural and Synthetic Fragrance Materials]

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Indian Standard

PERFUMERY MATERIAL — GERANIOL — SPECIFICATION

(Second Revision)

भारतीय मानक

सुगन्ध सामग्री – जेरानियोल – विशिष्टि

(दूसरा पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

January 1990 **Price Group 2**

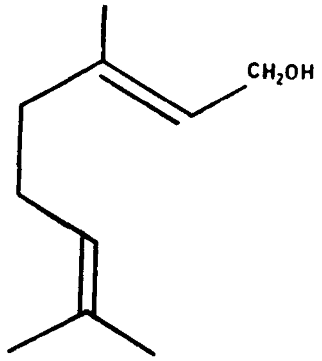
FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 25 June 1989, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1961 and subsequently revised in 1981, incorporating gas chromatographic method of analysis as an additional informative test method only. In the present revision, gas chromatographic analysis has been included as the main method of test for analysis.

At present, geraniol is made in the country primarily as ex-oil of citronella which in fact is not a single compound but a varying mixture of geraniol, citronellol, beta-elemene and nerol. The Committee responsible for the formulation of this standard felt that at times, there may be some difficulties in resolving such a mixture by GLC, and therefore, decided to retain wet method of analysis as an additional alternative method. However, for determining the minimum purity as total alcohols (that is geraniol, citronellol and nerol), GLC would henceforth be the main method.

Geraniol ($C_{10}H_{18}O$) is one of the most widely used perfumery chemicals for the compounding of all types of perfumes. Geraniol, an unsaturated primary terpene alcohol, is isomeric with linalool and nerol. It is represented by the following structural formula:



2-TRANS-3, 7-DIMETHYL-2, 6-OCTADIENE-1-OL
(MOLECULAR MASS 154·25)

Geraniol, both in its free state and in the form of esters, is an important constituent of several essential oils, namely, oil of palmarosa, oil of citronella and otto of rose.

There are many grades of geraniol available in the world market ranging from total alcohol content as low as 70 percent (geraniol for soap) to 98 percent (geraniol ex-palmarosa oil). Geraniol ex-natural source is mainly obtained by processing/fractional distillation of two main geraniol rich oils, namely, oil of citronella and oil of palmarosa. Geraniol ex-oil of citronella consists mainly of a natural mixture of geraniol and citronellol whereas geraniol ex-palmarosa is essentially geraniol. Geraniol is also produced synthetically from β-pinene, which besides geraniol, also contains varying amounts of other terpene alcohols, namely, nerol tetrahydrogeraniol and citronellol, depending upon the grade and efficiency, and economics of fractionation.

In India, currently geraniol from natural source only is produced on a commercial scale although attempts are being made to produce it synthetically. Hence for the purpose of this standard, only two types of geraniol, namely, geraniol ex-oil of citronella and geraniol ex-oil of palmarosa have been considered. Standards for geraniol ex-pinene will be incorporated as and when it becomes a commercial item in India.

(*Continued on third cover*)

Indian Standard

PERFUMERY MATERIAL — GERANIOL — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for the material commercially known as geraniol which is of the following two types:

a) Geraniol ex-oil of palmarosa, and

b) Geraniol ex-oil of citronella.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 1980	Relative density (*second revision*)
(Part 4) : 1980	Determination of optical rotation (*second revision*)
(Part 5) : 1986	Determination of refractive index (*second revision*)
(Part 6) : 1986	Determination of solubility (*second revision*)
(Part 8) : 1980	Determination of ester value, content of esters and combined alcohols (*second revision*)
(Part 9) : 1980	Determination of ester value after acetylation and free alcohols (*second revision*)
(Part 11) : 1986	Determination of carbonyl value and content of carbonyl compounds
IS 2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
IS 6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (*first revision*)

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 6597 : 1988 shall apply.

4 TYPES

4.1 There shall be two types of geraniol as given below:

a) *Type* 1 — Geraniol ex-oil of palmarosa, and

b) *Type* 2 — Geraniol ex-oil of citronella.

5 REQUIREMENTS

5.1 Description

The material shall be a clear liquid, free from sediment, suspended matter, separated water and adulterants when examined as prescribed in IS 326 (Part 2) : 1980.

5.2 Solubility

Both the types of the material shall be soluble in two or more volumes of ethyl alcohol (70 percent by volume) when tested by the method prescribed in IS 326 (Part 6) : 1986.

5.3 The two types of materials shall also comply with the requirements given in Table 1.

6 PACKING AND MARKING

6.1 Packing

The material shall be supplied in air-tight containers preferably amber coloured or opaque glass bottles, permitting a minimum air-space, as agreed between the purchaser and the supplier.

6.2 The material shall be protected from light and stored in cool and dry place.

Table 1 Requirements for Geraniol

(*Clause* 5.3)

Sl No.	Characteristic	Requirement		Method of Test, Ref to
		Geraniol Ex-oil of Palmarosa	Geraniol Ex-oil of Citronella	
(1)	(2)	(3)	(4)	(5)
i)	Colour and appearance	Colourless liquid	Colourless to pale yellow liquid	IS 326 (Part 2) : 1980
ii)	Odour	Sweet rosaceous	Characteristic rosaceous	IS 2284 : 1988
iii)	*Relative density at 27°C	0·876 5 to 0·879 5	0·871 5 to 0·884 5	IS 326 (Part 3) : 1980
iv)	Optical rotation	+ 0° 30′ to − 0° 30′	+ 2° to − 3°	IS 326 (Part 4) : 1980
v)	†Refractive index at 27°C	1·470 0 to 1·474 5	1·466 0 to 1·475 5	IS 326 (Part 5) : 1986
vi)	Total esters, calculated as geraniol, percent by mass, *Max*	1·0	1·0	Annex A
vii)	Total acetylizable compounds calculated as geraniol, percent by mass, *Min*	98	88	Annex A
viii)	Total carbonyl compounds calculated as citronellal, percent by mass, *Max*	—	1·0	Annex A

*The correction factor for relative density for each degree celsius change in temperature is 0·000 71.

†The correction factor for refractive index for each degree celsius change in temperature is 0·000 41.

6.3 Marking

The material shall be marked with the following information:

a) Name and type of the material;

b) Name of the manufacturer and/or recognized trade-mark, if any;

c) Net mass of the material; and

d) Batch number.

7 SAMPLING

7.1 Representative samples of the material, shall be drawn as prescribed in IS 326 (Part 1) : 1984.

8 TESTS

8.1 Tests shall be carried out as prescribed in **5.1**, **5.2** and the appropriate references specified in col 5 of Table 1.

8.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item No.* (vi), (vii) *and* (viii)]

GAS CHROMATOGRAPHIC ANALYSIS FOR GERANIOL

A-0 GENERAL

For determining ester content, total acelylizable compounds and total carbonyl compounds, GLC method would be the main method. In specific cases where difficulty is experienced in resolving mixture of components by GLC, ester content, total acetylizable compounds and total carbonyl compounds may be determined in accordance with IS 326 (Part 8) : 1980, IS 326 (Part 9) : 1980 and IS 326 (Part 11) : 1986 respectively.

NOTE — For determination of carbonyl compound, 5 gm of sample may be taken and proceeded by cold hydroxylamine method. For ester determinations 5 gm of the sample may be taken for test.

A-1 GAS CHROMATOGRAPHIC ANALYSIS FOR GERANIOL

A-1.1 Chromatographic conditions given here are for guidance only.

A-2 OUTLINE OF THE METHOD

A-2.1 A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-3 APPARATUS

A-3.1 Any gas chromatograph capable of being operated under conditions suitable for resolving individual constituents into distinct peaks may be used. The typical chromatogram for geraniol using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

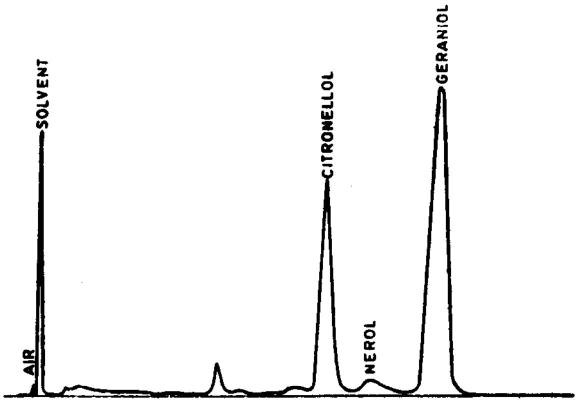


Fig. 1 Typical Chromatogram of Geraniol

A-3.1.1 *Column*

a) Material : Copper

b) Length : 5·49 m

c) Outer diameter : 0·635 cm

d) Inner diameter : 0·476 cm

e) Stationary phase : FFAP*, 10 percent by mass

f) Solid support Chromosorb WAW 60-80 mesh

A-3.1.2 *Carrier Gas*, hydrogen.

A-3.1.3 *Conditions*

a) Column temperature isothermal : 140°C

b) Injection port temperature : 200°C

A-3.1.4 *Detector*

a) Type Thermal conductivity

b) Temperature 285°C

A-4 CALCULATION

A-4.1 Area Measurements (*see* Note 1)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of the half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

A-4.2 Area Normalization (*see* Note 2)

By normalizing, it is meant calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTES

1 Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator if fixed with GLC machine would be of great advantage.

2 Internal standardization may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

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*The analysis may also be accomplished with columns containing: DEGS (diethylene glycol succinate) and FFAP (free fatty acid phase) in carbowax 20 M treated with nitrophthalic acid.

(*Continued from second cover*)

In the preparation of this standard, considerable assistance has been derived from the following publication:

EOA 16 Standard for Geraniol (Revised 1979), New York.

Essential Oil Association of USA.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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